Characterization Issues for Coated Conductor:

X-Ray Diffraction Techniques

OAK RIDGE NATIONAL LABORATORY

F.A. List, E.D. Specht, L. Heatherly, S. Cook

ARACOR (SBIR II)

J.A. Kerner

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At ORNL, continuous characterization is used routinely during conductor fabrication.

- Auger Electron Spectroscopy (*in situ*)

  - Surface Composition (~5 – 100 at. %)

- Laser Scatterometry (*ex situ*)

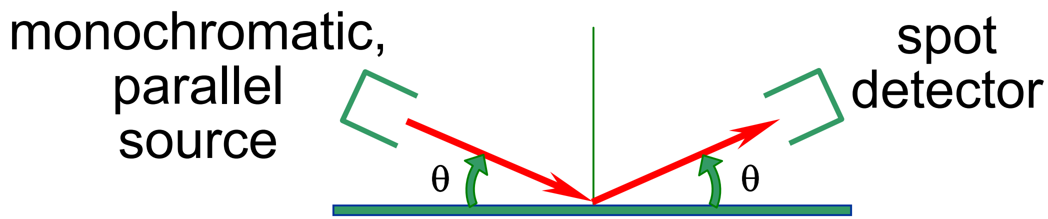
  - Optical Roughness (~2.5 – 1000 nm Ra)

- Parallel-Beam X-ray Diffraction (*ex situ*)

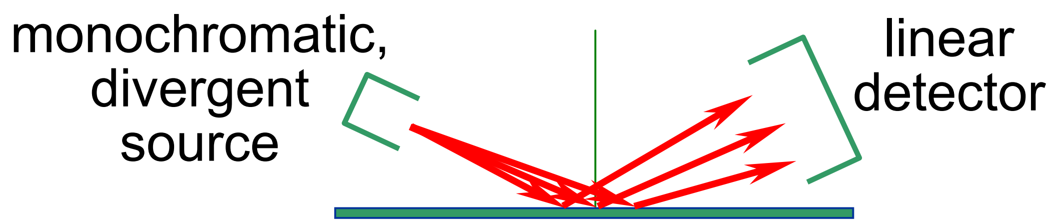
  - Crystalline Quality & Composition (θ-2θ, $\Delta\phi$, $\Delta\omega$, tape scans, pole figs.)

X-ray diffraction provides crystallographic information for epitaxial layers on textured substrates.

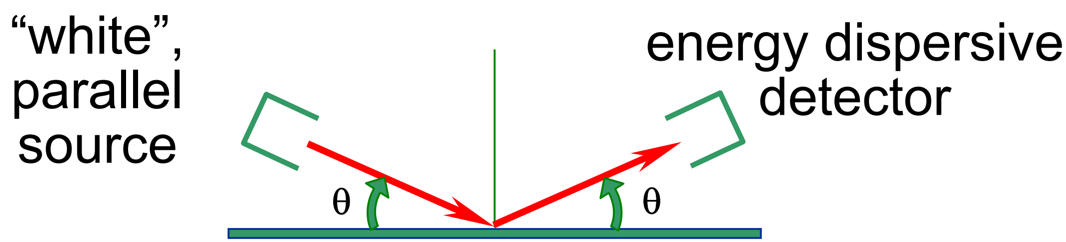
a) **Parallel-Beam** (**PBX**) Diffraction



b) **Divergent-Beam** (**DBX**) Diffraction

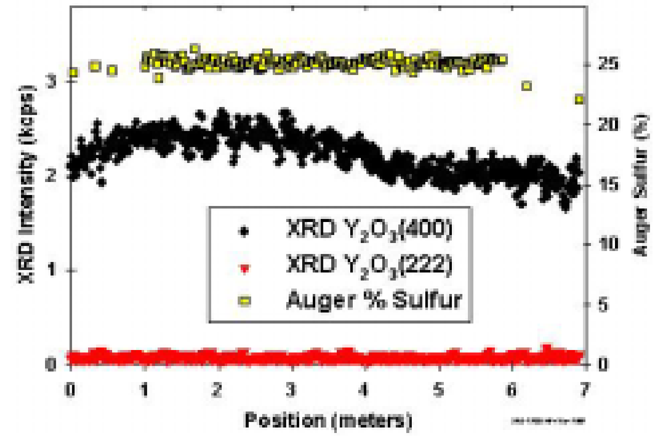


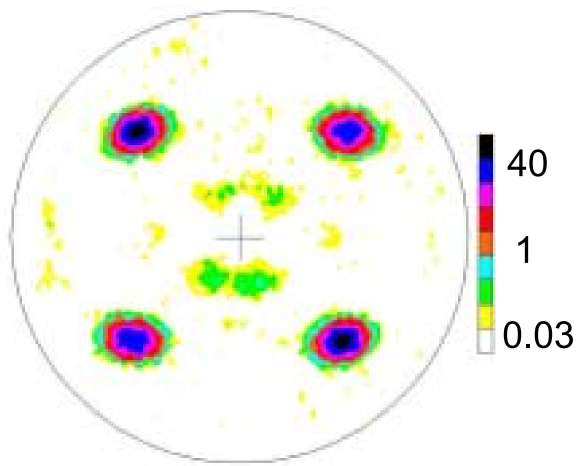
c) **Energy Dispersive** (**EDX**) Diffraction



At ORNL, two 4-circle PBX systems have characterized several kilometers of conductor.

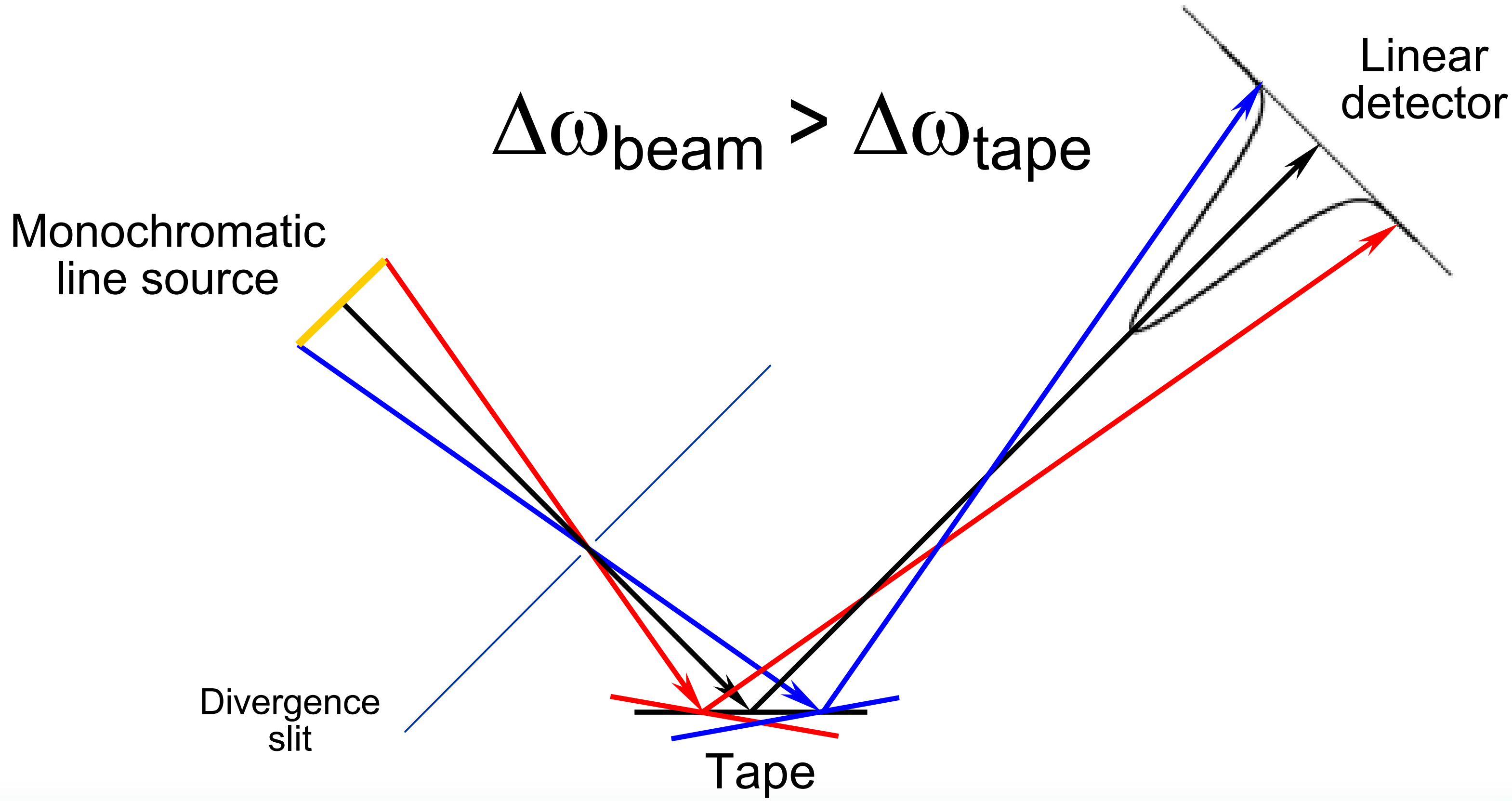
Y_2O_3(400)
Tape Scan
(7 meters)
(~7 minutes)



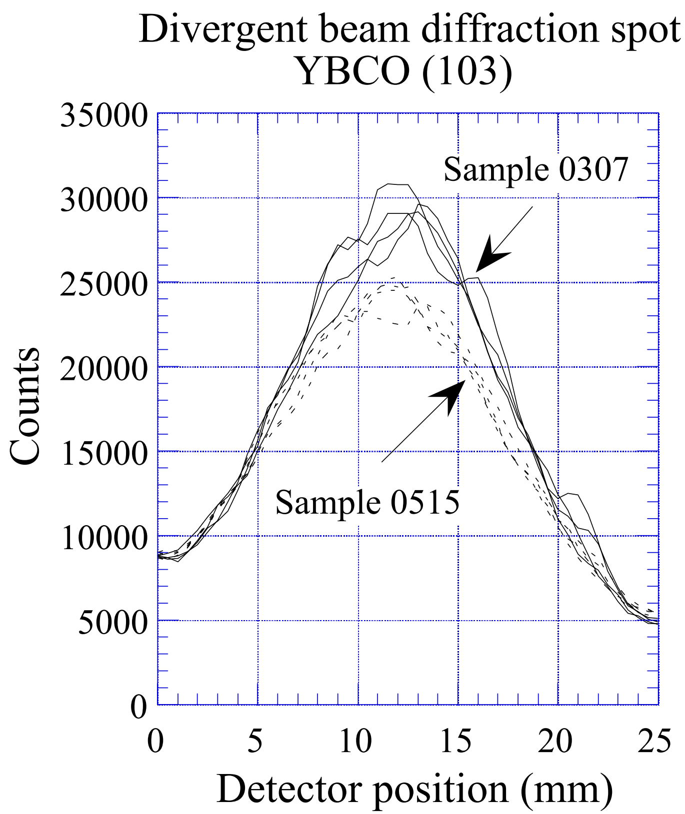


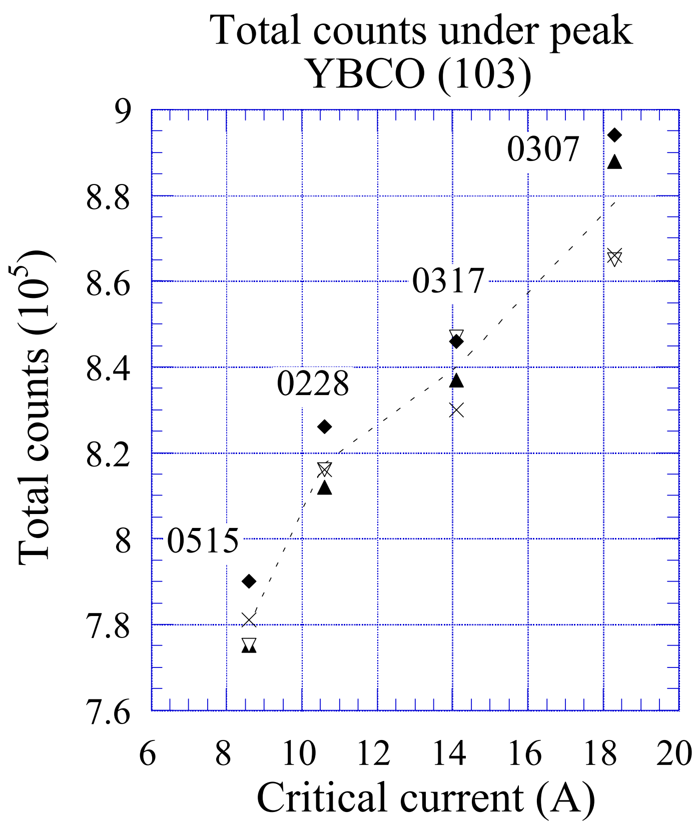
Ni(111)
Pole Figure
(~90 minutes)

For DBX, ranges of angle are simultaneously used for incidence and detection.

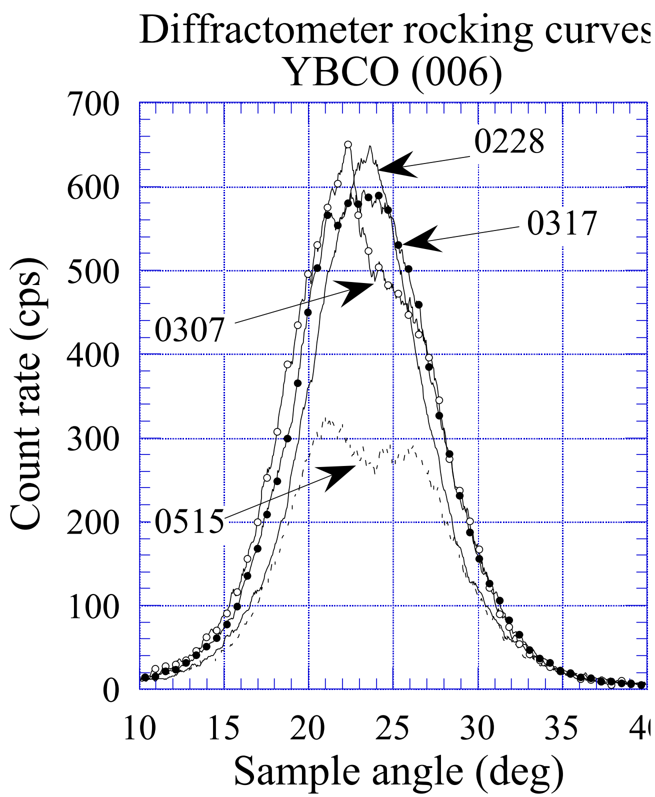


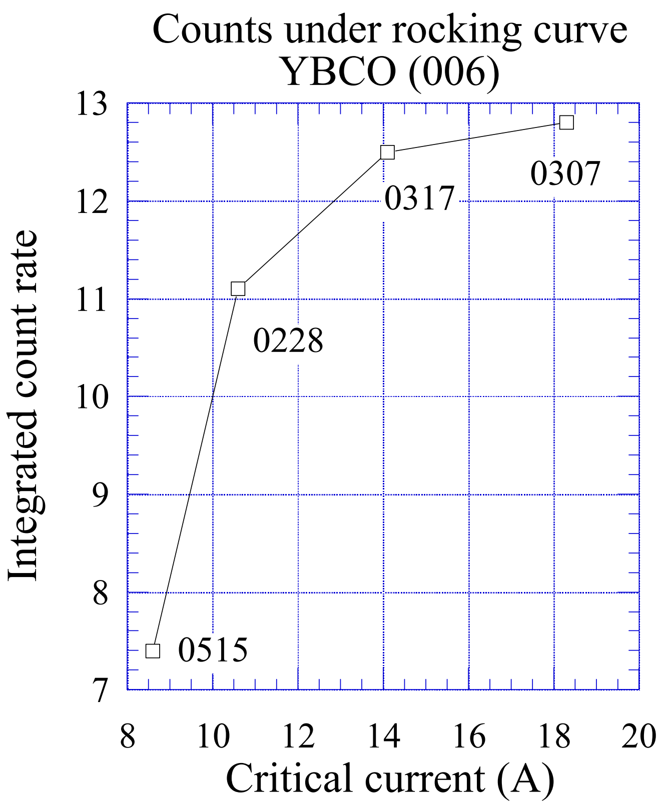
DBX data obtained at ARACOR correlate with critical current density.



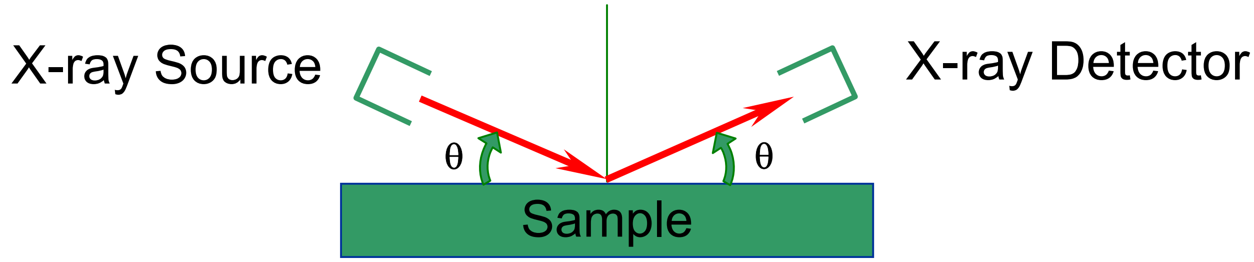


A similar correlation is observed for the same tapes using a PBX geometry.





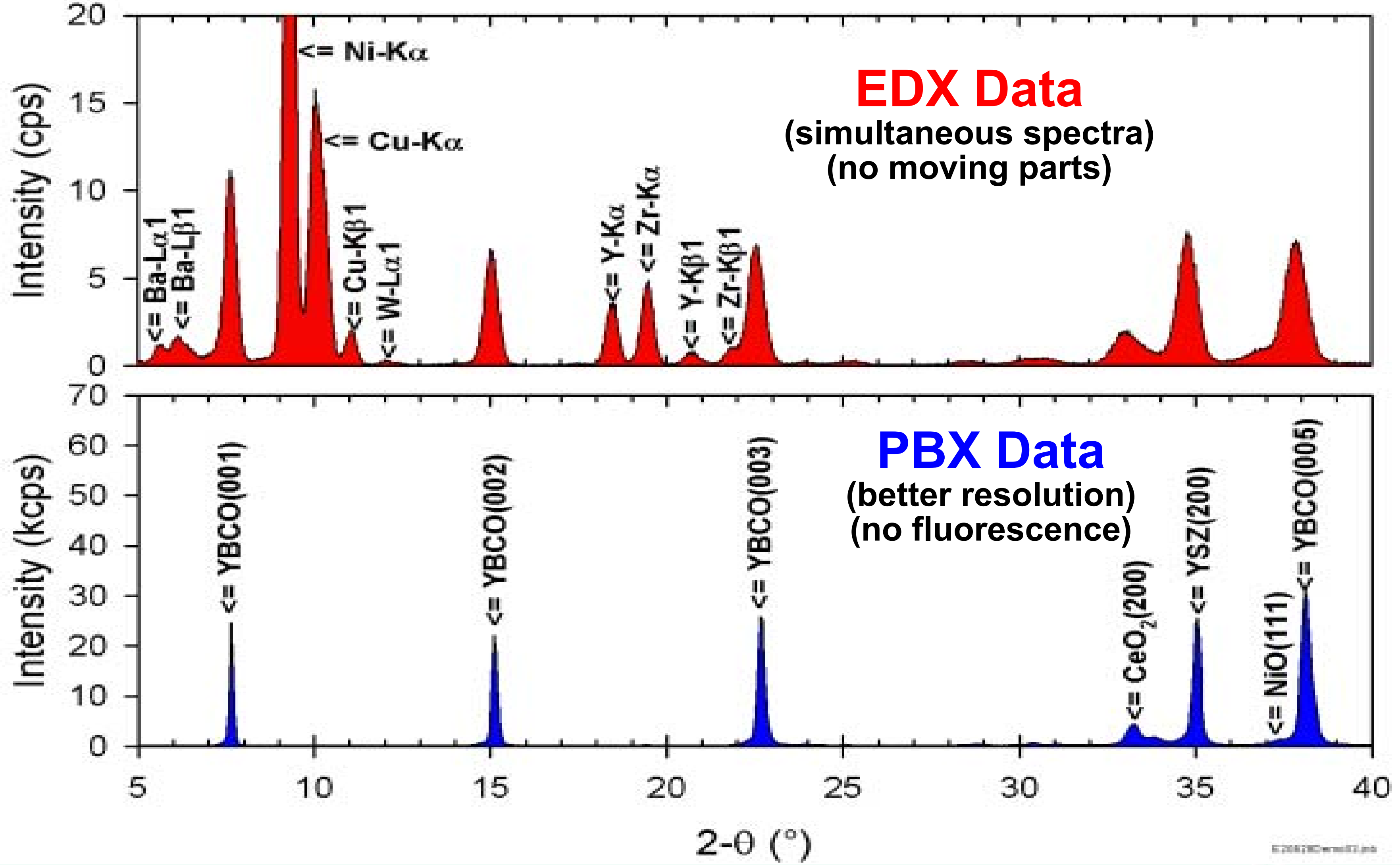
For EDX, ranges of energy (λ) are simultaneously used for incidence and detection.



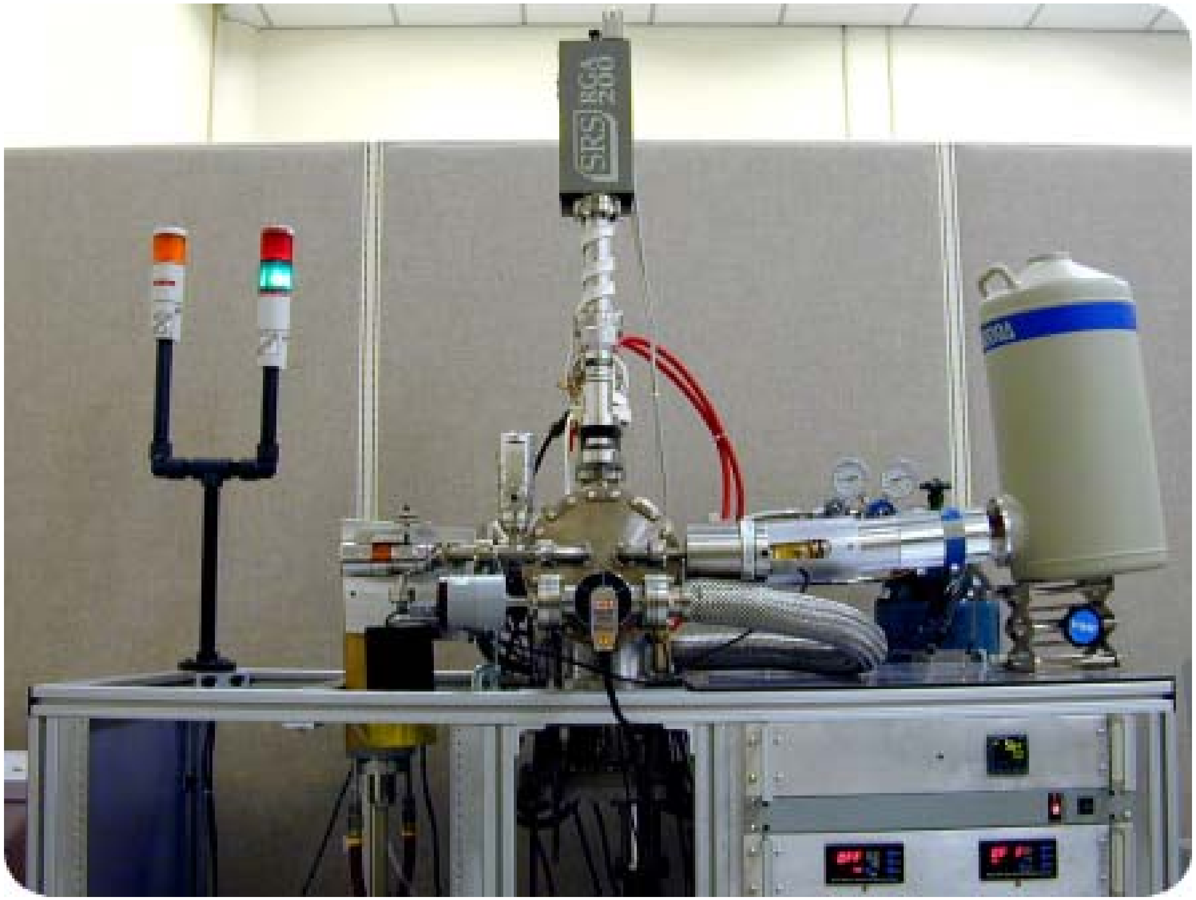
Bragg condition: $n\lambda = 2d \sin\theta$

Property	PBX θ-2θ geometry	EDX geometry
X-ray Source	Fixed λ (monochromatic Cu-Kα) (λ = 1.542Å)	Varied λ ("white" W) (λ = 0.2479Å to ∞ @ 50kV)
X-ray Detector	Varied θ	Fixed θ (=5°)
Data	I(θ)	I(λ or E)

EDX data are similar to PBX θ-2θ data.

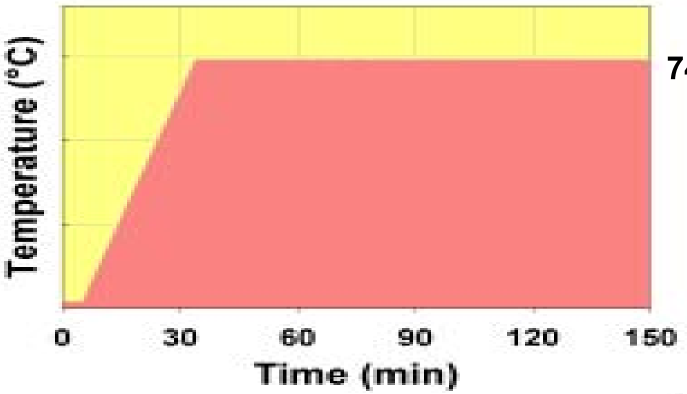


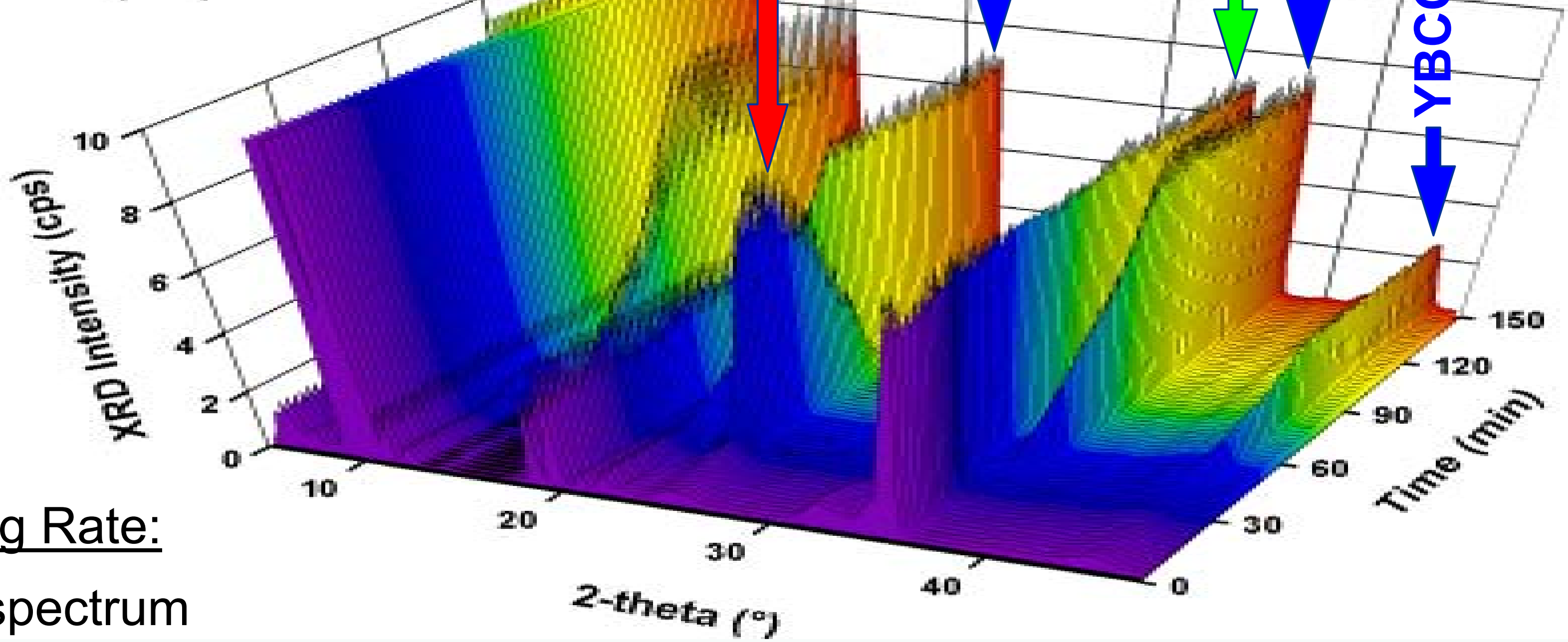
An EDX system has been used to study precursor conversion at ORNL.



- **Energy Dispersive X-ray Diffraction (EDX)**
  - "white" W source
  - EDX detector

A family of x-ray spectra clearly shows evolution of crystalline phases during conversion.





<u>Sampling Rate:</u>

2 min / spectrum

A comparison of properties for different diffraction geometries may suggest applications.

	Ex situ	*In situ* (candidates for process control)		
Attribute	**Parallel Beam (4-circle)**	**Parallel Beam (fixed beam)**	**Divergent Beam**	**Energy Dispersive**
Simplicity	Low	High	High	High
Flexibility	High	Low	Low	Low
Cost*	~$300k	~$65K	$70k	~$70k

* - approximate cost of prototype system

Complimentary data can be obtained using several diffraction geometries.

Attribute	Parallel Beam (fixed beam)	Divergent Beam	Energy Dispersive
Data	"Tape scan"	ω-scan or ϕ-scan	Full θ-2θ scan
Rate	~1 sec/point	~1 sec/scan	~100 sec/scan
Information	Crystalline phase content	Texture ($\Delta\phi$, $\Delta\omega$)	Crystalline phase content/distribution
Issues	Drift of d-spacing	Peak overlap	Resolution & fluorescence

Summary / Conclusion

- X-ray diffraction can provide measures of specific crystalline phases (d-spacings) having specific crystallographic orientations.
- A knowledge and control of crystallographic orientation is required for successful fabrication of epitaxial HTS coated conductors.
- Other important film properties include:
  - Non-crystalline components
  - Elemental composition
  - Stress state
  - Thickness
  - Non-epitaxial components
  - Chemical state
  - Morphology
  - Roughness